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IS 12614-2 (1988): Methods of Chemical Analysis of Ferro-molybdenum, Part 2: Determination of Total Carbon [MTD 5: Ferro Alloys]



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“ज्ञान एक ऐसा खजाना है जो कभी चुराया नहीं जा सकता है”

Bhartrhari—Nitiśatakam

“Knowledge is such a treasure which cannot be stolen”

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Indian Standard

**METHODS OF CHEMICAL ANALYSIS
OF FERRO-MOLYBDENUM**

PART 2 DETERMINATION OF TOTAL CARBON

भारतीय मानक

फेरो-मोलिब्डेनम के रासायनिक विश्लेषण की पद्धति

भाग 2 टोटल कार्बन ज्ञात करना

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FOREWORD

This Indian Standard (Part 2) was adopted by the Bureau of Indian Standards on 17 October 1988, after the draft finalized by the Methods of Chemical Analysis of Ferrous Metals Sectional Committee had been approved by the Structural and Metals Division Council.

IS 1559 : 1961 'Methods of chemical analysis of ferro-alloys' covers the chemical analysis of different ferro-alloys, namely, ferrosilicon, ferrochromium, ferromanganese and speigelleisen, silicomanganese, ferromolybdenum, ferrophosphorus, ferrotitanium, ferrovanadium and ferrotungsten. While reviewing this standard, the sectional committee had decided that a separate series for each ferroalloys be prepared. Accordingly, IS 1559 in its various parts was published for the chemical analysis of ferro silicon.

The chemical analysis of ferromolybdenum is covered in a series of parts of this standard. With the publication of these parts the analysis of relevant constituent prescribed for ferromolybdenum in IS 1559 : 1961 will be superseded. This part covers the method for determination of total carbon. The other parts are:

Part 1 Determination of molybdenum

Part 3 Determination of silicon

Part 4 Determination of sulphur

Part 5 Determination of phosphorus

Part 6 Determination of copper

Part 7 Determination of aluminium

In this revision the volumetric method for determination of carbon has been retained and updated and the gravimetric method has been deleted.

In reporting the result of a test or analysis made in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS 2 : 1960 'Rules for rounding off numerical values (revised)'.

*Indian Standard***METHODS OF CHEMICAL ANALYSIS
OF FERRO-MOLYBDENUM****PART 2 DETERMINATION OF TOTAL CARBON****1 SCOPE**

This standard (Part 2) covers the volumetric method for determination of total carbon in ferro-molybdenum.

2 REFERENCES

The following Indian Standards are necessary adjuncts to this standard:

<i>IS No.</i>	<i>Title</i>
IS 228 (Part 1) : 1987	Methods for chemical analysis of steels Part 1 Determination of carbon by volumetric method (for carbon 0.05 to 2.50 percent)
IS 266 : 1977	Specification for sulphuric acid (<i>second revision</i>)
IS 1472 : 1977	Methods of sampling ferro-alloys for determination of chemical composition (<i>first revision</i>)
IS 6226 (Part 1) : 1971	Recommendations for apparatus for chemical analysis of metals: Part 1 Apparatus for determination of carbon by direct combustion

3 SAMPLING

3.1 Sample shall be drawn and prepared in accordance with IS 1472 : 1977.

3.2 The sample is cleaned with organic solvents like, such as ether or acetone and dried in an air oven at $100 \pm 5^{\circ}\text{C}$ before use.

4 QUALITY OF REAGENTS

Unless specified otherwise analytical grade reagents shall be employed in the test.

**5 DETERMINATION OF TOTAL CARBON
BY DIRECT COMBUSTION
(VOLUMETRIC METHOD)****5.1 Outline of the Method**

The sample is burnt in a stream of pure oxygen,

and the resulting carbon dioxide and excess of oxygen are collected in a specially jacketed burette and the carbon dioxide absorbed in alkali. On passing the excess of oxygen back to the burette the decrease in volume is read against a scale, calibrated directly to the percentage of carbon.

5.2 Apparatus

Apparatus as given in IS 6226 (Part 1) : 1971 may be used.

5.3 Reagents

5.3.1 Potassium Hydroxide Solution, 30 percent (*m/v*)

5.3.2 Concentrated Sulphuric Acid, $\rho_d = 1.84$ (conforming to IS 266 : 1977)

5.3.3 Soda-lime, granules (0.80 — 2.0 mm)

5.3.4 Potassium Permanganate, solid

5.3.5 Dilute Sulphuric Acid, 2 percent (*v/v*)

5.3.6 Fluxes, pure tin and iron (99.99 percent pure)

5.4 Procedure

5.4.1 Weigh accurately 1 g of the sample in a zirconia based or alumina refractory boat. Add 0.5 g of (tin and iron) combined flux and introduce it into the heated combustion tube of the furnace heated to 1300°C .

5.4.2 Close the furnace inlet with a rubber stopper and let the boat attain the temperature of the furnace. Pass a stream of oxygen slowly (400 ml/minute) into the furnace and establish connection with the burette which has been previously filled with acidulated water so that the liquid level in the bulbed portion of the burette does not fall rapidly. After a minute or so, the level of water in the burette begins to fall more rapidly, though the same rate of flow of oxygen is maintained, indicating completion of the combustion. Slow down the rate of flow of oxygen. Take the reading when the level reaches near the zero graduation mark after closing the

bend way stop cock and equalizing the liquid levels of the burette and the connecting levelling bottle. Pass the collected and measured gas twice into the absorption pipette and when absorption is complete, measure the contraction in the volume.

5.4.3 A standard ferromolybdenum of known carbon content shall be used for standardizing the apparatus.

5.4.4 Blank

Change a pre-ignited boat in a furnace with the same amount of flux used in the determination

and follow the procedure as in 5.4.1 and 5.4.2.

5.5 Calculations

$$\text{Carbon percent} = (A - B) \times F$$

where

A = burette reading after absorption of carbon dioxide in potassium hydroxide solution;

B = burette reading for the blank; and

F = correction factor for temperature and pressure as given in Table 1 of IS 228 (Part 1) : 1987.

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